5. TEST AND MATERIALS STANDARDS

A. Rolling Contact Fatigue

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
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Objective

- Characterize the rolling contact fatigue (RCF) performance of ceramics and tribological coatings that are under consideration for use in rolling element components (e.g., hybrid bearings)
- Determine the effects of subsurface damage, properties such as (static and dynamic) hardness and fracture toughness, and the stress state from Hertzian contact on RCF performance.
- Model hypothetical microstructures that will promote improved RCF performance.
- Link RCF performances measured by different internationally used RCF test techniques.

Approach

- Establish three-ball-on-rod (3BOR) RCF test facilities and collaborations with domestic and international institutions that employ different RCF test techniques. Correlate test results.
- Vary machining-induced subsurface damage in a silicon nitride (Si_3N_4) and correlate that to measured RCF performance.
- Provide new understanding of RCF performance/microstructure relationship to ceramic manufacturers that will serve to promote microstructure engineering and improved RCF performance.

Accomplishments

- Established 3BOR RCF facility.
- Initiated formal collaboration with Bournemouth University (BU) in the United Kingdom to relate performances measured with 3BOR and three-ball-on-ball (3BOB) RCF test techniques.
- Defined machining conditions and designed and procured test coupons. Initiated testing.

Future Direction

- Conduct domestic interlaboratory study to compare 3BOR performances of a selected Si_3N_4.
- Compare RCF damage on rods and balls of a selected Si_3N_4 with the same coarse and fine grinding.
- Conduct international interlaboratory study to compare performances measured with different RCF test methods.

Introduction

The use of ceramics in rolling elements shows many practical advantages over traditional hard and bearing-grade steels. Current demands on load-bearing contacts in all kinds of machinery are leading to developments aimed at running them at high speeds with minimum vibration, hostile environments, increased unit loads, and restricted lubrication. The design and manufacture of such contacts are at the limit of established technology; hence the need for ceramic contacts.

Si_3N_4 is one class of ceramic that has combined properties that are most suited for rolling contact conditions. Experimental programs with Si_3N_4 contacts using full-scale and bench tests have produced some understanding of RCF modes failure and durability; however, the effects of grinding or finishing rate on the subsurface integrity and hence RCF durability and failure modes are poorly understood.

To address these issues, this project is characterizing the RCF performance of Si_3N_4 compositions that are under consideration for use (or that are presently used) in rolling element components (e.g., hybrid bearings, cam roller followers) (see Figure 1). There are several different test methods to measure RCF performance, so this project also works toward reconciling their measured differences so that their results can be validly pooled. The study of the effects of subsurface damage, properties such as (static and dynamic) hardness and fracture toughness, and the stress state from Hertzian contact on RCF performance is a natural outcome. Based on those findings, hypothetical microstructures that will promote improved RCF performance will be identified and shared with ceramic manufacturers.

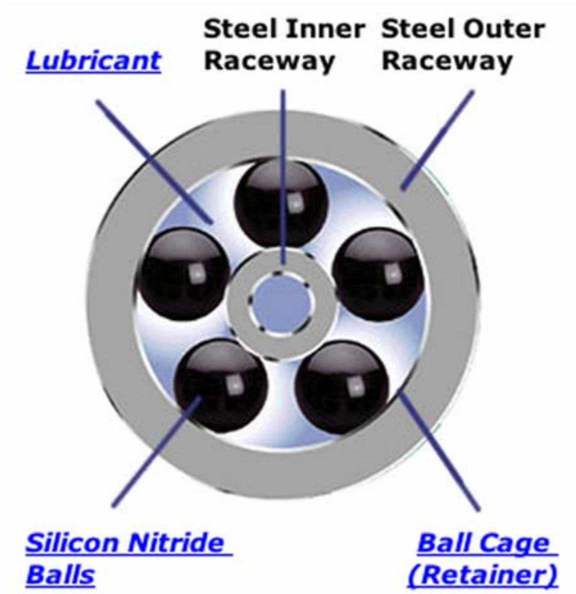

Figure 1. Schematic of a hybrid bearing system that includes silicon nitride balls. *Source: www.cerbec.com.*

Approach

There are three primary aspects to the project—measure RCF performance and reconcile results that were generated using different RCF test techniques; examine the effects that subsurface damage and microstructure have on RCF performance; and iteratively work with ceramic manufacturers and communicate RCF results and interpretations those manufacturers can then use to develop lower-cost Si_3N_4 compositions, improve Si_3N_4 machinability, improve RCF performance, or combinations thereof.

A multi-task, 3-year project involving BU and the Oak Ridge National Laboratory (ORNL) was initiated in the last quarter of FY 2004; this collaboration will serve as a key component of the project. A BU Ph.D. student will be a primary performer of these tasks and will alternate on-site research at BU and ORNL over the life of the project, spending about half of the time at each institution. This student will work under the supervision of Prof. Mark Hadfield at BU and the project principal investigator at ORNL.

RCF performance of Si_3N_4 will be interrogated using 3BOB and 3BOR (NTN–Bower) RCF test methods conducted at BU and ORNL, respectively; and RCF performance measured with each technique will be linked. Additionally, the generated subsurface damage in cylindrical rods and spheres machined under the "same" machining conditions will be compared to assess/verify the sought equivalence. Ceradyne's Ceralloy 147-31N will serve as the model Si_3N_4. A critical element of this project will be the identification, characterization, and interpretation of subsurface damage; these will be accomplished through a variety of inspection means and test techniques. Rotating flexure testing, optical and scanning electron microscope fractographies, dye penetration, optical coherent tomography, scanning acoustic microscopy, and residual stress analysis will be used. Machining damage in Ceralloy 147-31N, which was systematically characterized and exploited by uniaxial flexure testing in a recent study,[1,2] will provide a useful guide to RCF sample preparation. Indentation and scratch testing and finite element analysis (FEA) will also be used to supplement the characterization and interpretation of results. Finally, the student, project principal investigator, and Hadfield will integrate the results and interpret the effect of subsurface damage on RCF in Si_3N_4.

Results

RCF performance is being interrogated as a function of pre-existing subsurface damage in Ceralloy 147-31N Si_3N_4. Specimens were ground in one of three ways: 100-grit roughing and 600-grit finishing; 180-grit roughing and 600-grit finishing; and the conventional method for machining RCF specimens (1200-grit finish). Table 1 provides additional details of the machining.

The effects of subsurface damage in monolithic ceramics are critical to RCF performance, but that relationship has largely been taken for granted and is little understood. To help resolve the issue, numerous test coupons were prepared or designed to facilitate the quantification of a critical independent parameter in this project: depth of machining damage. Ceralloy 147-31N RCF (Figure 2), half-RCF (Figure 3), rotary bend strength (RBS) (Figure 4), half-RBS (Figure 5), and ASTM C1161B test specimens have been prepared and are undergoing testing. All five of those specimen geometries were machined according to the conditions outlined in Table 1. The RBS, half-RCF, half-RBS, and ASTM C1161B (uniaxial 4-point-bend flexure) specimens are being tested in parallel with the RCF specimens because flexure tests are efficient at quantifying the depth of subsurface damage (via companion fractography) and at exploiting the effects of sub-surface damage (on strength in this case). The RBS specimen rotates during its monotonic loading to fracture, so its entire gage section is subjected to outer-fiber tensile stresses. This will enable the study of strength-size scaling effects. The half-RCF and half-RBS specimens were strength-tested in four-point-flexure (Figure 6). The strength calculation for the half-RCF (i.e., half-cylinder) geometry is straightforward; however, the calculation of fracture stress for the half-RBS geometry from the fracture load is not. FEA of the half-RBS specimen (Figure 7) was used, and failure load was linked to maximum outer fiber tensile stress in the specimen's gage section. Weibull strength statistics for the half-RBS specimen are listed in Table 2. Strengths among the three sets were not appreciably different.

Table 1. Ceralloy specimens were prepared by one of three finishing conditions

Finish	Step	Wheel	Removal (in.)	Removal per pass (in.)
Coarse	1. Roughing 2. Induce damage 3. Finishing	Accepted practice 100 grit 600 grit	 0.004 0.0005	0.001 0.001 0.0001
Fine	1. Roughing 2. Induce damage 3. Finishing	Accepted practice 180 grit 600 grit	 0.004 0.0005	0.001 0.001 0.0001
RCF-conventional	Use the "accepted" practice for RCF test bar finishing (Ref PO# SB 1341-02-N-1669). Fine grinding using 1200-grit diamond, per Siddiqui e-mail of February 18, 2004			

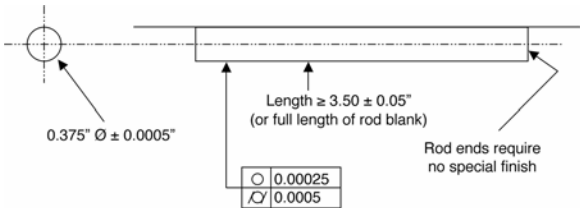

Figure 2. Schematic of RCF specimen geometry. This specimen is used in 3BOR RCF tests.

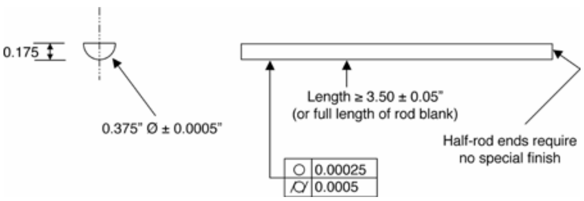

Figure 3. Schematic of half-RCF specimen geometry. The four-point-flexure flexure strength distribution and depth of machining-induced subsurface damage were measured.

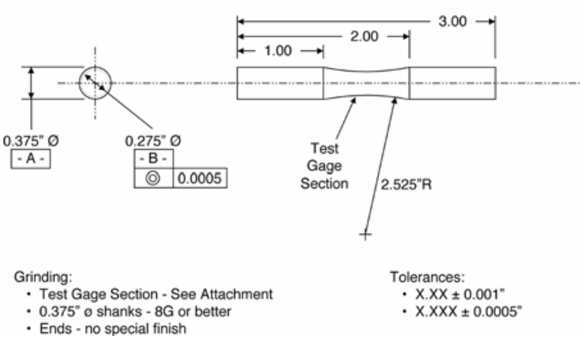

Figure 4. Schematic of RBS specimen geometry. This specimen is being subjected to rotary bending and monotonically loaded to fracture—a new test method developed in this program.

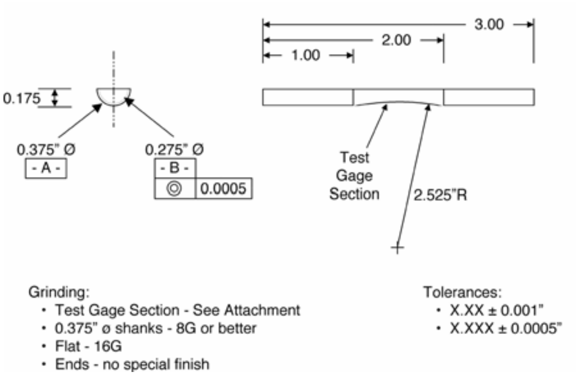

Figure 5. Schematic of half-RBS specimen geometry. The four-point-flexure flexure strength distribution and depth of machining-induced subsurface damage were measured.

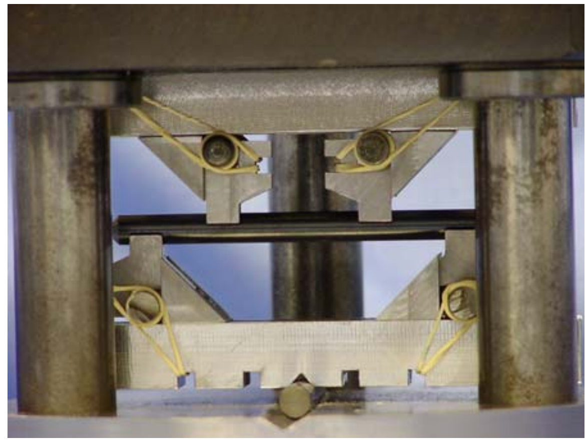

Figure 6. Four-point-bend fixture used to flexure-strength-test cylindrical or half-RCF or half-RBS specimens.

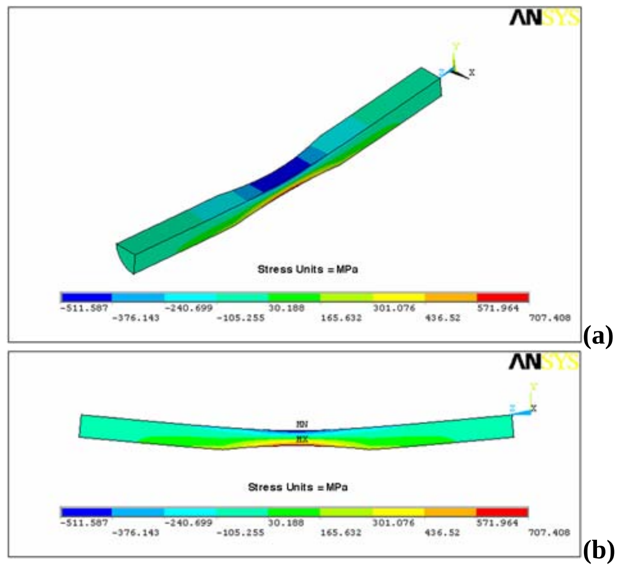

Figure 7. Oblique view (a) of FEA stress analysis of half-RBS specimen. Symmetry was used to simplify the model, so only one-half of the half-RBS specimen was analyzed. Four-point-flexure failure load was used to calculate the outer fiber tensile stress (b) that existed at fracture.

Fractography of these specimens (and all the other flexure specimens) is presently under way to examine the depth of machining-induced subsurface damage.

To better understand RCF damage and its link to material microstructure, several supplemental characterization test methods are used. For example, instrumented static and dynamic indentation testing and instrumented scratch testing of the Ceralloy

Table 2. Half-RBS flexure strength results

Finish	Number of test specimens	Average strength (MPa)	Standard deviation (MPa)	Characteristic strength (MPa)	Weibull modulus
Coarse	10	715.6	99.1	753.6	8.1
Fine	10	756.4	51.9	775.2	15.1
RCF-Conv.	10	732.0	79.6	765.1	11.3

147-31N are used to explore how contact loading damage (as a function of loading rate) is affected by sub-surface damage. The competition of quasi-plastic damage and cracking processes in Ceralloy 147-31N is being interrogated as a function of the depth of the sub-surface damage and compared with polished material as well. NBD200 Si_3N_4 (NIST standard reference material for Knoop hardness) and NC132 Si_3N_4 (NIST standard reference material for fracture toughness) are also being tested; specimens of those materials are presently undergoing indentation and scratch testing, the results of which will serve as a performance reference for comparing the performance of Ceralloy 147-31N and whatever additional ceramics are ultimately tested in this program. SN101C and TSN-03NH (both bearing grades of Si_3N_4) are undergoing instrumented indentation and scratch testing as well, and their performance will be compared with that of the other listed silicon nitride compositions. Raman spectroscopy, which can measure residual stresses, is being used. Preliminary results suggest that residual stresses (manifested by changes in wave number peak location in Figure 8) can indeed be quantified.

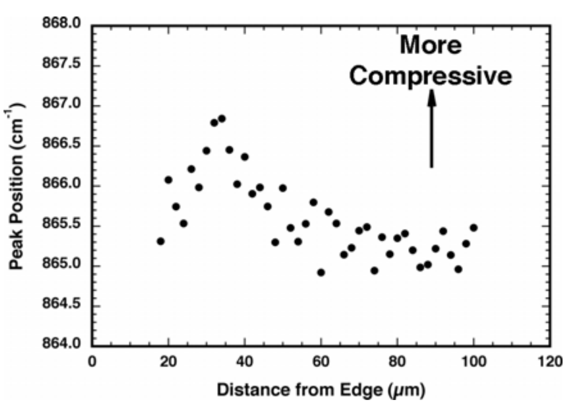

Figure 8. PiezoRaman spectroscopy profile of Raman peak position as a function of distance from the edge in a SN101C ball. Higher peak position is proportional to residual compressive stress, so this measurement shows that a gradient in compressive stress exists as a function of depth.

An interlaboratory study of research institutions and universities within the United States is being organized whereby RCF testing will be performed on samples of Si_3N_4 and the results combined and compared. Colorado State University and the Air Force Research Laboratory have so far given verbal commitments of participation.

In addition to the collaboration with BU, interactions exist with Germany's Bundesanstalt für Materilforschung und–prüfung, Japan's National Institute of Advanced Industrial Science and Technology, and the United Kingdom's National Physics Laboratory to compare RCF performance. These interactions exist under the auspices of the International Energy Agency Annex III. Working group participants met in January in Cocoa Beach, FL; the next meeting is tentatively set for June 2005 in Kobe, Japan, coincident with the International Tribology Conference.

Dialogue with ceramic bearing manufacturers is critical to maintaining relevance in this program. Frequent communication occurs between the principal investigator and chief scientists at several domestic manufacturers (Cerbec/Saint-Gobain, Ceradyne, Enceratec, Kennametal, and Cercom) regarding this project's test matrix, plans, and progress.

Conclusions

A 3BOR RCF test facility was established at ORNL, and the evaluation of ceramic RCF performance is now under way. A formal collaboration was initiated with BU for 3BOB RCF testing of ceramics; it will enable the eventual reconciliation of RCF performance measured with those two techniques. The effects of subsurface damage on RCF performance are being scrutinized. Several supplemental characterization routes (e.g., flexure strength testing and subsurface damage interrogation, damage evaluation from Hertzian indentation, piezoRaman spectroscopy) are used to assist in the interpretation of RCF performance. Domestic and international interlaboratory RCF studies are planned to compare and verify ORNL measurements with those generated at other laboratories and institutes. Finally, new under-standing of the RCF performance/microstructure relationship will be provided to ceramic manufacturers that will serve to promote microstructure engineering and improved RCF performance.

References

1. J. Kang, R. T. Cundill, and M. Hadfield, "The Consequences of Aggressive Lapping Processes on the Surface Integrity of HIPed Silicon Nitride Bearing Balls," *Tribology in Environmental Design 2000*, Professional Engineering Publishing, London.

2. G. D. Quinn, L. K. Ives, and S. Jahanmar, "On the Fractographic Analysis of Machining Cracks in Ground Ceramics: A Case Study on Silicon Nitride," NIST Special Publication 996, National Institute of Standards and Technology, U.S. Department of Commerce, May 2003.

Presentations

A. A. Wereszczak, "IEA Annex III Working Group Meeting on Rolling Contact Fatigue," presented at the 28th International Cocoa Beach Conference on Advanced Ceramics and Composites, Cocoa Beach Hilton, Cocoa Beach, FL, January 27, 2004.

A. A. Wereszczak, "ORNL Characterization of Ceramics for Armor, Transportation, and Energy Applications," presented at Cercom, Inc, Vista, CA, February 25, 2004.

A. A. Wereszczak, "ORNL Characterization of Ceramics for Armor, Transportation, and Energy Applications," presented at Ceradyne Inc, Costa Mesa, CA, February 26, 2004.

A. A. Wereszczak, "Evaluation of Ceramic Deformation Processes Through Hertzian Indentation," presented at Saint-Gobain, Worcester, MA, August 3, 2004.

B. Implementing Agreement for a Programme of Research And Development on Advanced Materials for Transportation Applications

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Contractor: Oak Ridge National Laboratory, Oak Ridge, TN
Prime DOE Contract Number: DE-AC05-00OR22725

Objectives

- Facilitate the integration of new technologies into the transportation sector by implementing research that validates the applicability of these technologies to improve material properties while maintaining acceptable life-cycle costs.
- Promote commercialization of new materials technologies by developing standard testing and characterization methods in conjunction with national and international standards communities.

Approach

- Define and implement research under the International Energy Agency (IEA) Implementing Agreement (IA) entitled *Implementing Agreement for a Programme of Research and Development on Advanced Materials for Transportation Applications* (IA–AMT).
- Conduct major research themes as annexes under the current IA:
  - Annex II: Co-Operative Program on Ceramics for Advanced Engines and Other Conservation Applications
  - Annex III: Co-operative Program on Contact Reliability of Advanced Engine Materials

Accomplishments

- Created a web site that provides the international technical community with information about (1) the mission and strategy of the IA, (2) details of the various annexes, and (3) a number of technical reports generated by collaborative research tasks conducted under Annex II. The site is at http://ia-amt.ornl.gov/index.html.
- Initiated Annex IV, A Cooperative Program on Integrated Engineered Surface Technology. Technical efforts associated with this annex are under way.
- Initiated Annex V, Light-Weighting of Materials. Planning efforts are under way.
- Canada and the United Kingdom joined the IA. Canada will participate in the lightweighting materials activity, while the United Kingdom will participate in Annex III.

Future Direction

- Develop plan for lightweighting materials annex and present to the Executive Committee for approval.
- Initiate test plan for Annex V.

Introduction

The current mission of the IA–AMT is to investigate promising new technologies for evaluating and ultimately improving the performance of materials for transportation systems. The primary motivation for this activity is the fact that new materials technologies are required to increase efficiency and reduce harmful emissions in these systems. Examples of these technologies include (1) lightweighting to improve fuel efficiency; (2) surface engineering to improve the resistance to wear and contact damage; (3) development of durable coating systems for thermal, wear, and environmental management; and (4) development of revolutionary materials (structural ceramics and ceramic matrix composites) for operation at much higher temperatures and pressures. As discussed in this report, the research activities within the IA–AMT focus specifically on (1) the identification of promising new technologies for improving materials performance and (2) the development of specialized characterization techniques for validating the applicability of this technology to improve material properties while maintaining acceptable life cycle costs.

At present the active contracting parties for the IA-AMT are

- Germany—Bundesanstalt für Materialforschung und –prüfung (BAM)
- Canada—Materials Technology Laboratory, CANMET
- United Kingdom—Department of Trade and Industry
- United States—U.S. Department of Energy

Approach

In the area of performance improvement, the current emphasis is on integrated engineered surface technology (IEST) and lightweighting of materials. IEST encompasses the synthesis, processing, characterization, and application of technologies that enhance the functionality of surfaces in contact with the environment or with the surfaces of other solids. Activities on lightweighting of materials focus on aluminum, high-strength steels, magnesium, metal and polymer composites, titanium, intermetallic alloys, and other advanced materials. Current topics under consideration include (1) data on production and resource availability; (2) life cycle data on environmental impacts associated with production, processing, and use of lightweight materials; (3) recycling information including regulatory frameworks; (4) data on crashworthiness, design, and testing methodologies; (5) data on base material cost; (6) data on energy impacts of lightweight materials; and (7) shared information on research programs on lightweight materials.

In terms of performance evaluation, the primary focus is on techniques for (1) assessing environmental degradation of structural (non-oxide) ceramics; (2) evaluating time-dependent degradation of the mechanical performance of structural ceramics; (3) quantifying key properties of coatings for wear, thermal, and environmental protection of current transportation materials; and (4) developing techniques for measuring key properties (topography, chemistry, subsurface damage) of engineered surfaces. Items 1 and 2 are motivated by the need to address key barriers to the use of this important class on material. For example, given the recent concern over environmental degradation of non-oxide ceramics in combustion environments, cost effective techniques are required to simulate these effects as well as to assess the effectiveness of environmental barrier coatings. The IA–AMT is currently evaluating a variety of techniques ranging from complex high-pressure burner rig tests to a simple cost-effective steam injection system. Ceramic coatings hold considerable promise for (1) improving wear resistance, (2) providing thermal protection, and (3) reducing environmental degradation of critical metallic components used in internal combustion engines. Unfortunately, techniques for assessing key properties, particularly with respect to the interface, are unproven. Item 3 addresses this limitation. In a similar fashion, as surface modification technologies mature, proven characterization techniques will be required to validate their performance (Item 4).

Results

The activities related to performance improvement and performance evaluation are covered in 4 annexes. Annex II focuses on pre-standardization (i.e., performance evaluation) issues related to the deployment of structural ceramics in transportation activities. Specific applications include ceramic diesel exhaust values, cam roller followers, timing plungers, etc. Results from these subtasks have led to the optimization of techniques for (1) characterization of powder properties, (2) quantification of the green-state characteristics, and (3) evaluation of mechanical performance. This extensive set of results generated in Annex II has been used in the establishment of standards [via the American Society for Testing and Materials (ASTM), Japan Industrial Standards (JIS), the Committee for European Normalization (CEN), the International Organization for Standards (ISO)] and National Institute of Standards and Technology (NIST) guidelines (see the publications list), which in turn have benefited the entire ceramics community.

Table 1 summarizes some of the existing standards that have benefited from the work conducted in this IA. In the case of room-temperature flexural strength, the test standard, ASTM C1161, was revised to reflect lessons learned about fixturing and test specimen configurations. ISO 14704 evolved from several standards (ASTM C1161, CEN EN843-1, and JIS R1601) and lessons learned from the IA–AMT work. The high-temperature flexural strength standard, ASTM C1211, evolved about the time of the IA–AMT round robin (Subtask 5–Annex II) and included lessons learned from C1161 and the IEA work. ISO DIS 17565 (not yet a standard) has evolved from several standards (ASTM C1211, CEN prEN820-1, and JIS R1604) and lessons learned from the IA–AMT subtask. The room temperature tensile strength standard, ASTM C1273, was developed as the IA–AMT results were being reviewed and analyzed and included many lessons learned. ISO 15490 evolved from two standards (ASTM C1273 and JIS 1606); and the IA–AMT round robin results were instrumental in establishing test specimen configurations, gripping arrangements, allowable bending, and test rates. The thermal shock standard, ASTM C1525, was developed after the IA–AMT subtask on thermal shock was completed. Although this standard follows a more conventional approach to thermal shock by using water quenching and standard MOR bars, insights garnered from the IA–AMT work are used in providing guidance to users in notes and discussions.

Current activities are focused on the evaluation and consolidation of nanocrystalline ceramics. Participants include the United States and Germany.

Table 1. Standards that have benefited from the IA subtasks

Property	**JIS**	**ASTM**	**CEN**	**ISO**
Flexural strength: RT	R1601-95	C1161-02	EN 843-1:95	14704:2000
Flexural strength: HT	R1604-95	C1211-98	prEN 820-1	DIS 17565
Statistical analysis	R1625-96	C1239-00	ENV 843-5:97	CD 20501
Fractography		C1322-02	prENV843-6	
Tensile strength	R1606-95	C1273-00		15490:2000
Sample preparation for the determination of particle size distribution of ceramic powders	R1619:95	C1282-00	EN 725-5:96	14703:2000
Surface area	R1626-96	C1274-00	EN 725-6:96	DIS 18757
Particle size distribution of powder by laser diffraction method	R1629:97			TC206 NP02
Thermal shock	R1615-93	C1100-98	prEN 820-3	
Flowability	R1639-4:99			TC206 WI93
Size distribution of granules	R1639-1:99			
Binder content of granules				
Drying loss of granules	R1639-3:99			
Bending fatigue: RT	R1621:95	C1368-00		TC206 PWI 07
Bending fatigue: HT	R16xx:01			

RT = room temperature; HT = high-temperature

Annex III also focuses on performance evaluation with emphasis on the evaluation of contact damage. International activities include a formal collaboration between Mark Hadfield of Bournemouth University (BU) in the United Kingdom and Oak Ridge National Laboratory (ORNL) in the United States. The study of the effects of subsurface damage (e.g., that results from component machining) on the rolling contact fatigue (RCF) performance of silicon nitride (Si_3N_4) is the primary theme. A BU Ph.D. candidate will serve as the cornerstone of the collaboration and alternate on-site research at BU and ORNL over the life of the project. The RCF performance of Si_3N_4 will be interrogated using ball-on-three-ball (Plint TE92) and three-ball-on-rod (NTN) RCF tests conducted at BU and ORNL, respectively. The Ph.D. candidate will attempt to link RCF performances measured with each technique. Additionally, the generated subsurface damage in cylindrical rods and spheres machined under the "same" mching conditions will be compared to assess/verify the sought equivalence.

A second international activity involves the preparation of a written report detailing techniques on RCF testing in each of the participating countries. Participants include Japan (AIST, KOYO-Seiko, and NSK), Germany (BAM), United Kingdom (NPL), and the United States (ORNL). The report will be completed at the end of 2004.

In 2005 and 2006, RCF studies involving both international and domestic participants will be implemented. Details of this international activity will be developed at the next IEA Annex III RCF Working Group meeting, tentatively set for June 2005 in Kobe, Japan—coinciding with the International Tribology Conference. Attendance of participants from Japan and the United Kingdom has been confirmed. The primary goal is to reconcile differences in the various test techniques and hardware. Results, including recommendations, will be forwarded to the appropriate standards setting organizations.

The effort conducted within the United States consists of an interlaboratory study of RCF testing involving research institutions and universities. Colorado State University and the U.S. Air Force Research Laboratory have so far given verbal commitments of participation.

Annex IV, A Cooperative Program on Integrated Engineered Surface Technology, focuses on the implementation of a vertically integrated engineered surface concept: combining surface texturing, thin films, and lubrication to create an engineered surface appropriate for different applications, with an overall objective of reducing friction and increasing durability. Although engineered surfaces can play a dominant role in energy conservation and utilization, several technical challenges must be addressed before such technology can be fully utilized. One broad area of crucial importance and mutual benefit is the development of international standards and practices for textured surfaces under different application conditions. Characterization and mechanical properties of an integrated engineered surface in terms of friction reduction, load-bearing capability, fatigue mechanisms, and life prediction are important issues that need to be addressed. Activities under Annex IV shall include various forms of characterization, evaluation methods, modeling, bench-scale testing, and demonstration field trials.

The implementation of lightweighting strategies in the transportation sector represents a major approach to increasing fuel efficiency and reducing emissions of greenhouse gases. Accordingly, a number of countries (see Table 2) are involved in extensive research activities to develop and validate advanced materials and manufacturing technologies for significantly reducing vehicle body and chassis weight. Materials under consideration include high-strength steels, magnesium, aluminum, titanium, metal-matrix composites, plastics, and ceramics, where applicable. Annex V, Light-Weighting of Materials, focuses on methods required for both performance improvement and evaluation, which are of interest to the participants. Current topics under consideration include (1) data on production and resource availability; (2) life cycle data on environmental impacts associated with production, processing, and use of lightweight materials; (3) recycling information, including regulatory frameworks; (4) data on crashworthiness, design, and testing methodologies; (5) data on base material cost; (6) data on energy impacts of lightweight materials; and (7) shared information on research programs on lightweight materials. In terms of recent achievements a draft plan outlining the scope of work, which covers areas of mutual interest, was prepared for discussion at the executive committee meeting in October 2004. Participants included the United States, Canada, and Australia (membership pending).

Table 2. Summary of lightweighting materials programs

Country	Program
United States	FreedomCAR and Vehicle Technologies Program *http://www.eere.energy.gov/vehiclesandfuels/technologies/materials/index.shtml*
Canada	AUTO21 Network of Centres of Excellence *http://www.auto21.ca/materials_e.html* Canadian Lightweight Materials Research Initiative (CLiMRI) *http://climri.nrcan.gc.ca/default_e.htm*
Australia	Light Metals Action Agenda *http://www.industry.gov.au/content/itrinternet/cmsindexpage.cfm* Cooperative Research Centre for Cast Metals Manufacturing (CAST) *http://www.cast.crc.org.au/*

Conclusions

The IA–AMT has made significant progress in expanding its scope, as evidenced by the addition of two new annex (IV and V). Both annexes are expected to generate new members. Given this progress, a formal request has been made to the IEA to extend the IA–AMT for a period of 3–5 years.

Publications/Presentations

Implementing Agreement for a Programme of Research and Development on Advanced Materials for Transportation Applications (IA-AMT), Strategic Plan, March 2004 (available at http://ia-amt.ornl.gov/.

International Energy Agency Implementing Agreement for a Programme of Research and Development on Advanced Materials for Transportation Applications, *Annual Report 2003*, February 2004 (available at http://ia-amt.ornl.gov).

C. Testing Standards

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Objective

- Develop mechanical test method standards in support of the Propulsion Systems Materials Program. New methods and sound engineering data will facilitate adoption of new materials in heavy vehicle propulsion systems.

Approach

- Conduct pre-standardization research on test methods that need refinement, or develop new test methods.
- Develop draft recommendations for practices or procedures based upon the needs identified by the research.
- Conduct round robins as necessary.
- Standardize procedures in the American Society for Testing and Materials (ASTM) and/or the International Organization for Standards (ISO).

Accomplishments

- Completed pre-standardization work on the flexural strength testing of split cylinders. A report is in preparation. Completed and fully documented all work on the fractographic characterization of grinding damage cracks.
- Completed a series of papers and made presentations.
- Completed about half of a NIST *Guide to Practice on Fractographic Analysis of Brittle Materials*.
- Revised, refined, and improved a variety of current ASTM and ISO standards.
- Helped found a new ASTM subcommittee on applications standards.
- Worked to correct and refine an ASTM standard specification for silicon nitride bearing balls.

Future Direction

- Write up the split cylinder test method and present the results to ASTM Committee C-28, Advanced Ceramics, for review. The error analyses for flexural strength testing of cylindrical rod specimens will be completed and a draft standard prepared for ASTM.

- Complete *the Guide to Practice on Fractographic Analysis.*
- Resume pre-standardization testing on the diametral compression strength test with the goal of standardization.

Introduction

This project creates new test methods that will facilitate the use of advanced materials in heavy-duty propulsion systems. Much of the work is for brittle materials such as ceramics, for which classical mechanical test methods developed for metals are not suitable. For example, tension-strength test specimens of many ceramic materials made in short, stubby cylindrical shapes (e.g., diesel engine fuel injector pins, timing plungers, valves) and classical dog bone shapes are impractical (Figure 1). Our goal is to adapt or refine existing test methods or invent new ones that will allow engineers and researchers to measure mechanical properties with good accuracy and precision. Formal test method standards are our primary objective. Sound test methods and high-quality databases will enhance the credibility of new materials and encourage engineers to use them in advanced heat engines.

Figure 1. Silicon nitride bearings.

Approach

Over the course of this program, we have formulated or contributed to the development of 17 ASTM and ISO standards. These have covered a range of topics including hardness, flexural strength, fracture toughness, fractography, elastic modulus, and Weibull strength distribution parameter determination. We have even contributed to a ceramic material specification for silicon nitride ball bearing materials. A spin-off benefit of this work has been that the generic test method standards have been used to create several biological ceramic materials specifications for dental and hip joint implant ceramics. Work continues on new alternative test methods.

A major element of this program has been the refinement of fractographic techniques to facilitate the detection and characterization of strength-limiting flaws in both laboratory-scale test specimens and components. The strength of ceramic components or parts is controlled by microscopic flaws, which may either be intrinsic to the manufacture or be introduced during grinding and finishing.

We also continued our work on several new mechanical test methods that are designed to measure mechanical properties of cylindrically shaped components or test coupons. Formal standard test methods are on the books for rectangular bend bars or tension-strength specimens, but these are not suitable for cylindrical parts (Figure 2). Simple methods for cylindrical parts are needed. Three methods we are working on are split cylinder flexural strength tests, rod flexural strength tests, and diametral compression strength tests.

Figure 2. Round specimens.

Important refinements and corrections were made to several existing standards during the past year. Thus "standards maintenance" activity is necessary to incorporate improvements and lessons learned from this and other projects.

Results

A major task was completed that involved fractographic characterization of grinding-induced cracks on the surface of finished ceramics. Figure 3 shows an example of a grinding crack in a rod

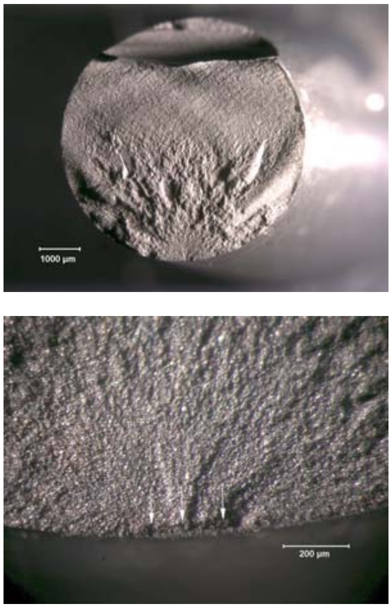

Figure 3. Fracture surface of a broken ceramic rod test specimen. The fracture origin is in the smooth semicircular region on the bottom of the figures. The origin is a "V-machining" crack (arrows in the lower figure) created when the rod was ground to final dimensions. Similar cracks can limit strength in components

strength test specimen. Several major reports and technical papers were prepared and presented to distribute this important information about grinding-induced damage. Lessons learned were incorporated into several ASTM standards.

An ASTM standard for fractographic analysis, C 1322, has already been adopted; but a more user-friendly *Guide to Practice for Fractographic Analysis* is also in preparation. The formal standard outlines the optimum procedures that engineers and fractographers should follow in performing fractography, but it assumes the analyst has moderate experience. The *Guide to Practice* will be a tutorial that will help the lay engineer learn the art of fractographic analysis of brittle materials and help transform what may seem to be a mysterious art into a routine engineering practice. It will have hundreds of example fractographs. Figure 4 is an example illustration showing how grazing incident angle lighting can help bring out fracture markings on fracture surfaces. The new *Guide to Practice* will include a set of recommendations for measuring

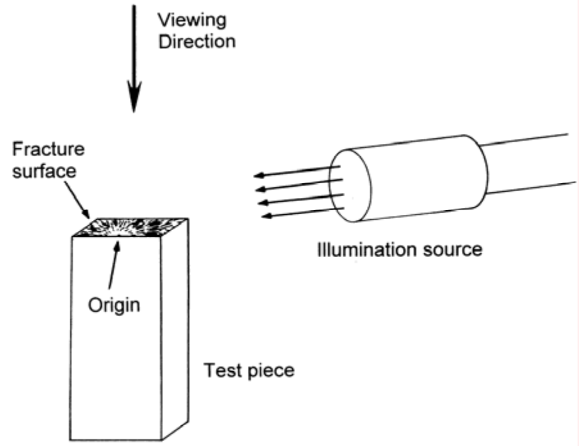

Figure 4. An example illustration from the *Guide to Practice for Fractographic Analysis of Brittle Materials*. This schematic shows vicinal, or low angle grazing illumination, which can create shadows that highlight features on the rough fracture surface of a broken test piece such as shown in the previous figure. Fractography can be effectively applied to understand the causes of fracture in the laboratory and in service components.

fracture mirror sizes. Fracture mirrors are relatively smooth zones surrounding fracture origins in ceramics, and their size can be directly correlated to the stress in the specimen or component at the instant of fracture. Fracture mirror analysis is powerful tool for forensic analysis since it can calculate the fracture stress even if the mode of loading is unclear or unknown.

Substantial progress was made this year on pre-standardization for the split cylinder strength test method. Short, stubby cylindrical parts are difficult to test for strength. A simple solution is to split the cylinder lengthwise and test the halves in a modified common flexural strength test fixture, as shown in Figures 5 through 7. Figure 8 shows a fracture surface of one of the rods. We had difficulty identifying the precise nature of the flaws in this material, but we eventually deciphered several fracture origins. A paper is in preparation on our findings. The Weibull strength distribution for one particular batch of material is shown in Figure 9. The findings of this work will be presented to ASTM Committee C-28, Advanced Ceramics, for review for suitability as a standard test method. Results will also be coordinated with the appropriate ceramic part manufacturers and engine companies.

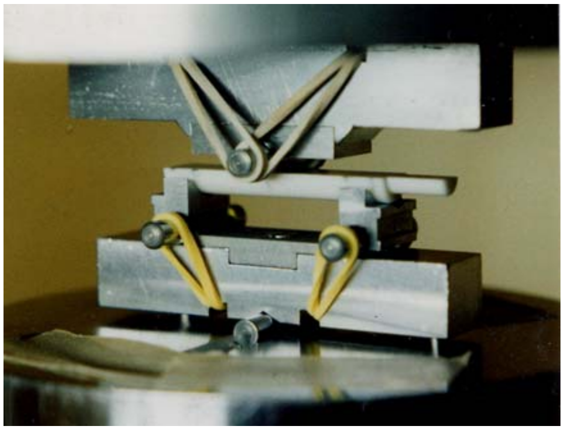

Figure 5. A ceramic split cylinder specimen (white) in a common three-point flexure strength fixture. This is a simple way to measure the strength of the ceramic part, which would be difficult to do for the intact part due to the high forces that would be necessary to break it. The finished round outer part surface is tested, and the cut surface which is on the top (compression side) of the bending loading, does not affect the results.

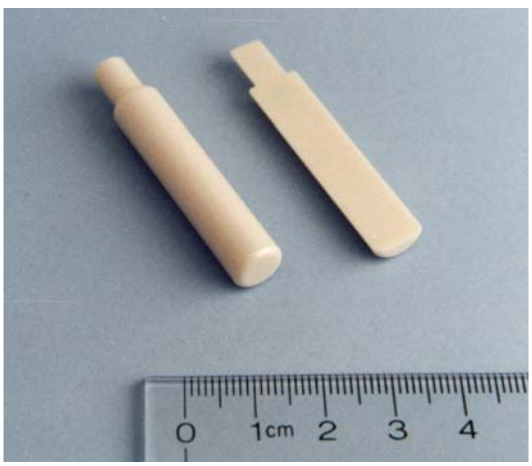

Figure 6. A cylindrical zirconia ceramic engine part. The part is a fuel injector pin for a heavy duty diesel engine. The left shows the intact part and the right shows a part cut in half. The latter is easier to test for strength.

Figure 7. Expanded view of the bend fixtures for a split cylinder (white specimen). Cradles hold the bottom of the specimen and distribute the load evenly. Load is applied to the specimen top (the cut surface) by the middle roller which is shown held in place by rubber bands on the subassembly on the top right of the photo. The middle roller is free to articulate or rock to match the specimen surface. Extra parts are shown in the fore and background.

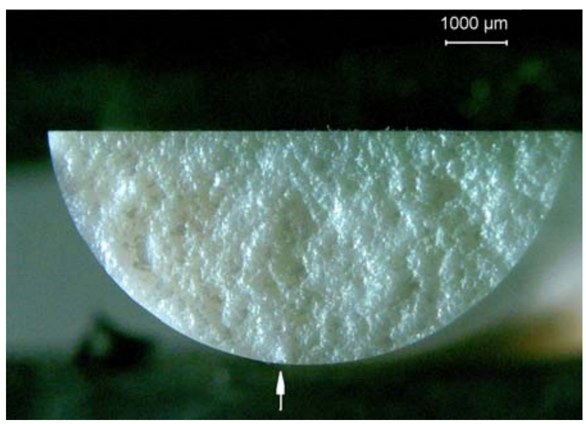

Figure 8. Optical fractograph showing the fracture surface of a broken split cylinder zirconia specimen. The arrow marks the fracture origin. Fractographic analyses was difficult in this material which had very rough fracture surfaces.

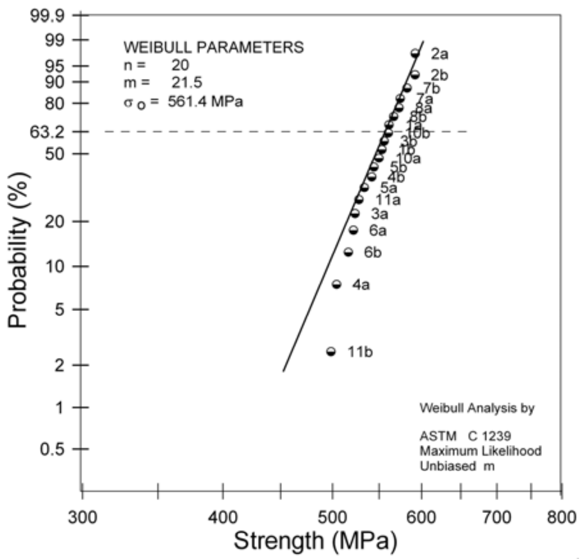

Figure 9. Weibull plot for split zirconia fuel injector pin parts.

We have made substantial progress in refining testing methods for measuring the flexural strength of full cylindrical rod specimens. Figure 10 shows a full-size rod specimen which, unlike the parts shown in earlier figures, is longer and therefore can be tested more easily. Cradles must be used to apply the forces evenly to the specimen. In FY 2004, we made progress on the analytical error analysis for this configuration. We expect to finish this work in the upcoming year, paving the way for standardization.

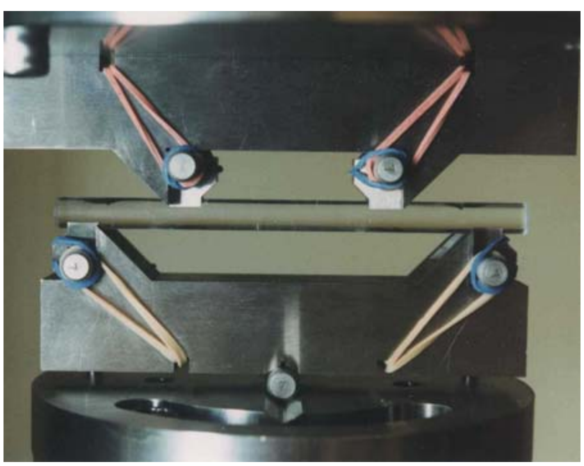

Figure 10. Flexural strength test fixture for a rod specimen.

Pre-standardization work on the diametral compression strength test specimen is planned for the upcoming year (Figure 11). Small pill-shaped specimens are loaded on their rims and split by tensile stresses. This is a convenient testing configuration, which in principle is ideally suited for small cylindrical parts; but there are some unsolved problems. We have completed some preliminary work and a review of the literature, which has revealed some of the problems with the method.

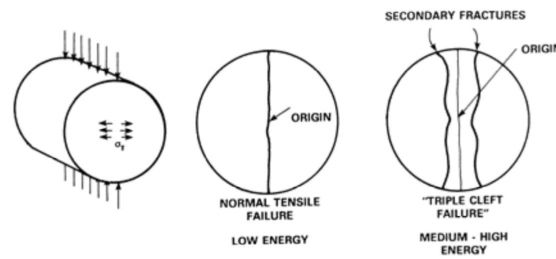

Figure 11. Diametral compression strength test.

Finally, we refined and updated a number of the standards created earlier in this program, including ASTM hardness, C 1326 and C 1327; ASTM fracture toughness, C 1421; ASTM fractographic analysis, C 1322; ASTM silicon nitride bearing specification, F 2094; and ISO 14704 and ISO 17565 on flexural strength at room temperature and high temperatures, respectively. As new information becomes available or shortcomings are revealed, it is prudent to update and correct these formal standards.

Conclusions

We have made progress in expanding the suite of test methods available for evaluating the properties of brittle ceramic materials. Our goal is to improve the test method and data base infrastructure that facilitates the incorporation of new advanced materials in heavy-duty diesel engines. Substantial progress was made in improving fractographic methods for detecting grinding-induced cracking and in developing test methods for measuring the strength of cylindrical parts. ASTM and ISO standards have been adopted, refined, and updated.

Publications/Presentations

G. D. Quinn, L. K. Ives, and S. Jahanmir, "Fractography Reveals Machining Cracks," *Bul. Amer. Ceram. Soc*. **82(**7) 11 (2003).

G. D. Quinn, L. K. Ives, and S. Jahanmir, "Machining Cracks in Finished Ceramics," to be published in *Fractography of Advanced Ceramics, II*, ed. J. Dusza, TransTech Publishing, Zurich, 2005.

G. D. Quinn, L. K, Ives, and S. Jahanmir, "Machining Damage Cracks: How to Find and Characterize Them by Fractography," pp. 383–394 in *Ceram. Eng. Sci Proc*. **24**(4), 2003.

G. D. Quinn, R. J. Gettings, and L. K. Ives, "A Standard Reference Materials for Vickers Hardness of Ceramics and Hardmetals," to be presented at the IMEKO Hardness Conference, National Institute of Standards and Technology, November 11–12, 2004.

G. D. Quinn, J. Eichler, U. Eisele, and J. Rödel, "Fracture Mirrors in a Nanoscale 3Y-TZP," *J. Amer. Ceram. Soc*. **87(**3) 513–516 (2004).

A. B. Kounga Njiwa, T. Fett, J. Rödel, and G. D. Quinn, "Crack Tip Toughness Measurements on Sintered Reaction-Bonded Si_3N_4," *J. Amer. Ceram. Soc*. **87(**8) 1502–1508 (2004).

D. Surface Modification of Engineering Materials for Heavy Vehicle Applications

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
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Subcontractor: National Institute of Standards and Technology, Gaithersburg, Maryland

Objectives

- Organize an international cooperative research program on integrated surface modification technology under the auspices of the International Energy Agency (IEA).
- Design and identify surface features and patterns that can achieve friction reduction and enhanced durability for heavy duty diesel engine components.
- Develop the understanding and appropriate models to explain the texturing effects on frictional characteristics. Develop appropriate thin films and coatings to achieve a synergistic and complementary relationship with texturing to enhance performance.
- Discover and develop a surface chemistry for protective films and coatings that works in synergy with the coatings.

Approach

- Determine the effect of size, shape, pitch, and patterns of surface textural features on friction under (1) a high-speed, low-load regime; (2) a high-load, high-speed regime; and (3) a high-load, low-speed regime.
- Develop cost-effective fabrication technologies for creating surface textural features on various surfaces including metals, ceramics, and coatings.
- Develop a test methodology to measure the effects of the textures on friction.
- Conduct research to develop an integrated system approach to combine the best practices in thin films, coatings, and surface chemistry for performances unrealizable by an individual approach alone.
- Concurrently, organize an international cooperative research program under the auspices of the IEA to pool resources and share this energy conservation technology worldwide.

Accomplishments

- Developed a new design principle for surface texture design to enable friction reduction under boundary lubrication conditions.
- Developed in situ instrumentation to allow direct observation of interface flow patterns with and without surface textural features.

- Visited several countries to establish agreement to join the IEA cooperative research activity.
- Visited various domestic companies and universities to organize a U.S. working group on integrated surface technology.
- Obtained final approval from the IEA Executive Committee on a new Annex on integrated surface technology in March 2004.

Future Direction

- Organize a U.S. national working group under the IEA banner to conduct joint research and provide information for international exchange.
- Organize an international working group and arrange a meeting at a location and time convenient to all participants.
- Add a modeling component to determine why some textural features function and some do not. The results will lead to design guidelines.
- Initiate the synergistic effect of thin films and coatings to protect textures.
- Explore the tribochemistry of various thin films and coatings.

Introduction

Frictional losses are inherent in all mechanical components in relative motion. The ability to control friction enables many technologies. Over the years, materials, lubricants, and surface modifications have provided the means to control and reduce friction to enhance energy efficiency in automotive and truck applications. A new avenue is needed to continue to increase the efficiency of energy utilization. Recently, dimples have been demonstrated to achieve friction reduction and durability enhancement in conformal contacts such as seals. The effect of dimples depends strongly on pattern design, contacting materials, and lubricant properties. A full understanding of these dependencies has yet to be developed. This research opens up a new avenue for increasing energy efficiency by combining surface textures, thin films and coatings, and the associated chemistry to protect surface textures. The objective of this project is to identify critical pattern features that control friction under a broad range of contact conditions and to develop models to explain these relationships.

Concomitant to this research, international cooperative research under the auspices of the IEA is planned to pool resources worldwide to accelerate this technology development. The results of this cooperative research will impact energy conservation efforts worldwide. Toward this end, the United Kingdom, Germany, Finland, Sweden, Israel, and Japan have agreed to participate in this activity under IEA Annex IV.

Approach

During FY 2004, experiments were conducted to examine various surface textures on steel surfaces using photolithography and chemical etching. Using the same area coverage (% of area occupied by the surface textural features), surface features such as grooves, triangles, ellipses, and circles were compared under high-speed, low-load conditions similar to those experienced by surface seals. Results indicated that (1) surface feature shape had great influence on friction reduction, and (2) friction reduction also depended strongly on the orientation of the surface features with respect to the direction of sliding. The results suggested that the conventional theory of hydrodynamic lift is not adequate to explain these observations, and a new theory is needed. Therefore, an in situ observation of the lubricant flow pattern will be developed to observe how a specific feature would affect the friction. A modeling effort will also be initiated in FY 2005.

To achieve energy savings, friction reduction needs to be achieved over a wide range of contact conditions existing in gears, transmissions, and engines. Therefore, the approach was to move into higher and higher loads and slower and slower speeds. New instrumentation and methods needed to measure the effect of textures will be developed.

New design principles for textural features will need to be developed to meet the load and speed requirements.

Results

Circular dimples were used to conduct high-load experiments using stainless steel specimens. The load was raised to 93 N on a smaller sample pin so that the apparent contact pressure was approximately 15–20 MPa. To prevent wear of the dimples, a fully formulated synthetic lubricant was used. Baseline specimens with and without dimples were compared. In each case, the dimples caused high friction. Grooves also exhibited higher friction when the contact pressure was increased or the speed reduced. These results tended to confirm the fluid mechanics models. A new design principle was needed to move into GPa contact pressure range.

Considering the basic principles of hydrodynamic lift, we designed a surface feature with a sloped bottom to provide an artificial hydrodynamic wedge. The idea was that if sufficient contact pressure were exerted, the surface would undergo elastic and plastic deformation and the liquid inside the feature would be squeezed, producing reactionary pressure to support the load. Since the pin-on-disk apparatus was limited by load, a four-ball wear tester with a configuration of ball-on-three-flat was used to generate high contact pressures. A 2.4-mm-diam 52100 steel ball bearing was used to slide on top of three 6-mm-diam disks. The testing conditions were 0.19–1.9 m/s speed and 2-, 5-, and 8-Kg loads lubricated by a fully formulated engine oil. The disks were also 52100 steel without case hardening. The surface texture was fabricated using mechanical scribing and electrochemical etching to create various sizes and shapes and slopes at the bottoms of the features. Since earlier work suggested that orientation with respect to the flow direction was a factor, the disks were carefully marked along the major axis of the features and mounted in the test apparatus. Figures 1 and 3 display the data in a 3-dimensional plot showing friction as a function of speed and load for steel on copper.

Figure 1 shows the baseline friction as a function of load and speed. Figure 2 shows the sliding direction in a typical hydrodynamic wedge shape; the friction coefficients across the speed and load range were lower than in the untextured case.

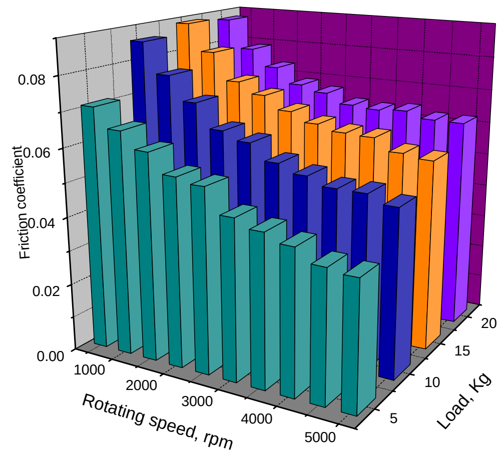

Figure 1. Untextured surface showing friction as a function of load and speed.

Figure 3 shows the sliding direction from shallow to deeper slope; the friction showed uniform improvement across the ranges. This finding was surprising, but it suggested other mechanisms might be in involved in addition to the classical hydrodynamic wedge theory. For these experiments, the contact pressure was approaching the GPa range. Instead of increasing friction, the hidden wedge geometry was able to reduce friction compared with the base case under boundary lubrication conditions. The dependence of the orientation direction was also surprising. If the major mechanism was to squeeze the fluid film along the wedge, then we would expect that flow direction along the long axis of the feature would give lower friction. For that matter, the cavitation mechanism would also suggest that this geometry would be better. The fact that flow along the short axis gives better friction control would seem to suggest that back-flow within the feature might play a more significant role than previously suspected.

Conclusions

We have demonstrated a new design principle that can reduce friction under boundary lubrication conditions. Additional tests on softer materials, in which we had more control over shaping the features, showed experimentally that a friction reduction of an order of magnitude was achievable.

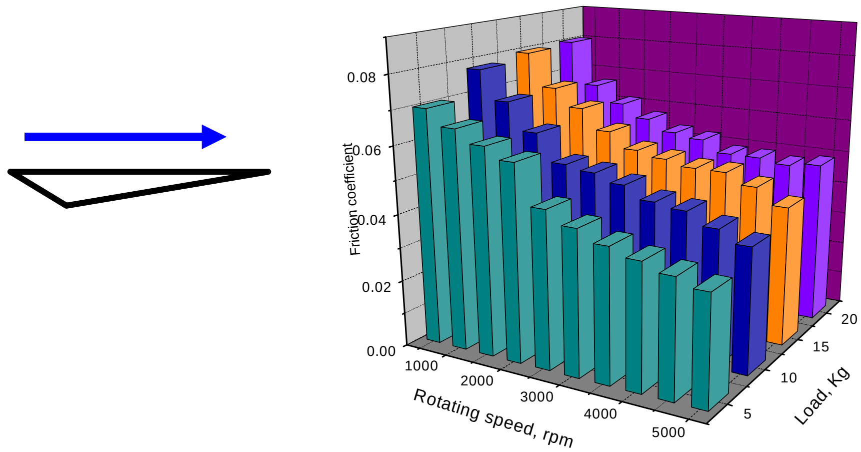

Figure 2. Textured surface with the flow toward the long axis of the feature.

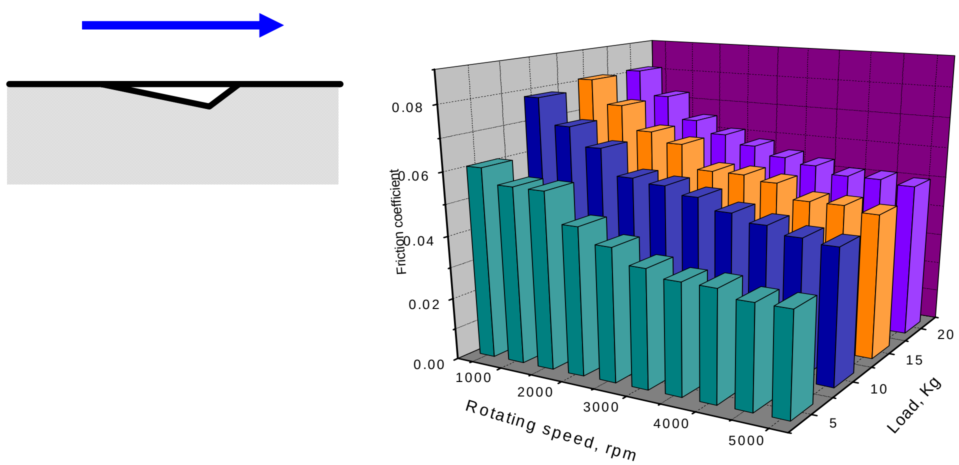

Figure 3. Textured surface with the flow toward the short axis of the feature.

This opens up a whole new avenue—the use of surface texturing in energy-intensive applications. The lack of an existing theory to adequately explain these observations suggests a new theory is needed. Therefore, in FY 2005, we will initiate a new modeling effort to increase understanding of the fundamental mechanisms.

Presentations

S. M. Hsu, "An Integrated Approach to Design Intelligent Surfaces for Heavily Loaded Contacts," presented at the International Joint Tribology Conference, Ponte Vedra, FL, October 27–29, 2003.

S. M. Hsu, "An Integrated Surface Texture Design with Surface Modification Techniques and Thin Lubricating Films," presented at the Smart Surfaces in Tribology: Advanced Additives and Structured Coatings Conference, Zurich, Switzerland, September 10–12, 2003.

S. M. Hsu, "An Integrated Surface Technology International Program under IEA," presented at the International Energy Agency Executive Committee meeting, Oakland, CA, October 20–23, 2003.

S. M. Hsu, "Surface Texturing under Boundary Lubrication for Friction Control," presented at the STLE annual meeting, Toronto, Canada, May 17–20, 2004.

Jorn Larson Basse, X. Wang, L. Ives, and S. M. Hsu, "Some Friction Experiments with Textured Surfaces," presented at the Nordic Symposium on Tribology, Troms, Norway, June 2004.

Jorn Larson Basse, X. Wang, L. Ives, and S. M. Hsu, "Some Friction Experiments with Dimpled Surface Texture," presented at the Fourth China International Symposium on Tribology, Xian, China, November 8–11, 2004.

S. M. Hsu, "An Integrated Surface Modification Technique to Control Friction: A New Paradigm," keynote speech at the Fourth China International Symposium on Tribology, Xian, China, November 8–11, 2004.

Y. Chae, X. Wang, and S. M. Hsu, "The Size Effect of Surface Texture on Lubricated Friction," presented at the First International Conference on Advanced Tribology, Singapore, December 1–3, 2004.

Publications

Jorn Larson Basse, X. Wang, L. Ives, and S. M. Hsu, "Some Friction Experiments with Textured Surfaces," in *Proceedings of Nordic Symposium on Tribology*, Troms, Norway, June 2004.

X. Wang and S. M. Hsu, "An Integrated Surface Modification Technique to Control Friction: A New Paradigm," keynote paper, in *Proceeding of the Fourth China International Symposium on Tribology*, Xian, China, November 8–11, 2004.